

INFORMATION REPORT

PREPARED AND DISSEMINATED BY

CENTRAL INTELLIGENCE AGENCY

COUNTRY

Hungary/East Germany

SUBJECT

Proposed Pilot Plant for Production of
Octyl-alcohol Utilizing Oxo-synthesis
Process/East German Oxo-synthesis Process
at I G Farben Works

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SUPPLEMENT TO REPORT #

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1.

the Hungarian Gas and Oil Research Institute

assigned

the task of developing plans for, and setting in operation, an oxo-synthe-
sis plant for the commercial production of octyl-alcohol ($C_7H_{15}CH_2OH$).
The general plans for this pilot plant, as proposed by the Ministry for
Chemical Industries, called for the utilization of high pressure reactor
units which were located at a former synthetic rubber plant in Rakoske-
resztur $[47 28 N - 19 15 E]$, about 10 mi east of Budapest. The respon-
sibility of installing more specific instrumentation and developing a
practical processing method was delegated

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2. As was often the case in ventures of this type, bureaucratic red tape and
budgetary problems plagued the operation from the outset. Begun in 1952,
the pilot plant had logged only two hundred operating hours as late as
1955 and never produced octyl-alcohol in marketable quantities. The spe-
cific underlying factor responsible for the plant failure was a financial
one involving the high cost of transporting to the plant, the large quan-
tities of CO and H gas necessary for our processing methods.
3. The problem of transporting input materials to the Rakoskerekasztur loca-
tion was never satisfactorily solved and in 1956 the plant was closed down
with the plan of setting up a completely new operating in Pappkeszi $[47 05 N -$
 $18 05 E]$. The proximity of Pappkeszi to Petfurdo $[47 09 N - 18 07 E]$, it
was felt, would enable the new plant to utilize Petfurdo's more accessible
CO and H gas supply, thus resolving what had been one of the major diffi-
culties. Future development plans even called for the laying of a pipeline
direct from Petfurdo to the plant location.
4. Engineering designs for the Pappkeszi pilot plant were drawn up in mid-1956
and budget requests for the proper instrumentation were submitted to the

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Ministry for Chemical Industries. [] intent to start construction on the plant early in 1957. Once again, administrative fumbling in the Ministry and reduced budgets slowed the project to a standstill. []

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[] in March 1957, the operation was still in the planning stage and construction had not begun. []

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5. In the event that the pilot plant's construction is ever accomplished []

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[] the basic plant and instrumentation data would be as follows:

- a. The high pressure reactors would be of a type developed at the Csepel steel works, employing the Schierenbeck process of manufacture. The reactor []

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would be approximately 30 ft high with an inside diameter of one and one-half ft. Lined with stainless steel, the reactor could withstand pressures up to 4300 lbs per sq in and temperatures up to 200°C. It was hoped that eventually []

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[] develop a reactor 42 ft high with an inside diameter of three and one-half ft with the same pressure and temperature capacities as the smaller model.

- b. The planned capacity of the pilot plant was 800 m/t of octyl-alcohol per year, with an expanding capacity to reach 1200 m/t. Three of the larger reactors mentioned above would be employed in an operation of this size. Plant electrical power would undoubtedly come in off the national grid on a 10 thousand v line.

6. The proposed budget for this plant, as requested in 1956, was for 40 million forints. [] this estimate would have to be revised upward by another 10 to 15 million forints should the project be started again within the near future.

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7. [] at peak operating capacity, a pilot plant of the size [] proposed would employ approximately 40 plant technicians; four plant engineers; one chemical engineer; and one or two chemical researchers for laboratory programs.

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8. For purposes of comparison, it should be noted that a pilot plant of this size and production capacity would be considered small by US standards. Several petro-chemical companies in the US carry on operations of this type in pilot plants which, in Hungary, would be large enough to supply most of the country's product need. The Hungarian petro-chemical industry suffered and will probably continue to suffer from a situation which saw competent technicians hamstrung by lack of facilities and adequate finances--as well as unrealistic administration by Ministry members who were purely political figures with little or no technical background.

Oxosynthesis Pilot Plant in Leuna-Merseburg

9. [] an opportunity to discuss with East German technicians, an oxosynthesis process as it was being carried on in early 1957 at the I G Farben Works located in Leuna-Merseburg.

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10. The East German process differed from the Hungarian method in that the East Germans concentrated on producing commercial alcohols through the use of synthetic hydrocarbons, whereas the Hungarian method utilized crude oils.
11. The Farben technicians exhibited more than a passing interest in the Hungarian methodology due to the fact that the Farben process did not include the continuous recovery of cobalt from the reactors--a fact which caused their operation to be much more expensive and time-consuming than our own.

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12. [] no opportunity to discuss the Farben plant operation or capacity at great length, but [] the East Germans [] had produced only test quantities of high molecular weight alcohol for use in plasticizers and detergents. Their 1956 production did not exceed 20 m/t.
13. Since both the East German and Hungarian processes for synthesizing alcohols depended on raw materials indigenous to the respective areas, coal on the one hand, crude oil on the other, I could foresee no changing of processing techniques by either scientific group. The East Germans felt that while their own system was more expensive than that employed by the Hungarian chemical industry, it best suited their facilities and purposes.
14. One other noteworthy fact concerning the development section of the Farben works in Leuna-Merseburg was the very small proportion of professional technicians to plant labor. The ratio of professional engineers and experienced technicians to skilled and unskilled laborers at the East German Farben plant was one technician to 70 laborers, as opposed to a ratio of one to 18 in West German Farben complexes. The underlying explanation for this situation is, of course, the more lucrative salaries offered to scientific personnel in the Western European countries.

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In the course of the conversion into aldehydes other products are also formed, including 10—20 per cent of the corresponding alcohols. This fact of considerable practical importance has only been investigated recently. *I. Wender* and co-workers have proved [2, 3], that these alcohols are formed in a homogeneous catalytic reaction from the primary products, the aldehydes. The catalyst, probably cobalt carbonyl hydride HCo(CO)_4 , formed from the cobalt compounds used, is sulphur resistant and active only at high carbon monoxide partial pressures. The reduction needs somewhat higher temperatures than the Oxo-process (the above mentioned investigators carried out their experiments at an average temperature of 180—185 °C) but the other reaction conditions are identical. In this manner it has become possible to convert n-butyraldehyde into n-butylalcohol with a yield of 72%.

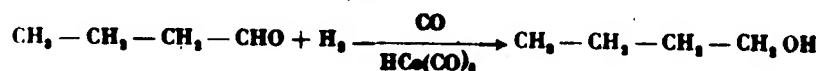
On the basis of the above, the Oxo-synthesis and the subsequent reduction can be carried out in one step in the operation named «direct alcohol synthesis». Starting from octene-1, a mixture of isomer nonyl alcohols has been produced in one step (*ibid*) with 61% yield.

G. Natta and co-workers [4] also investigated the direct alcohol synthesis. Working without hydrogen in the presence of secondary alcohols, they succeeded in getting a product which consisted of 70 per cent alcohols. The one-one mole hydrogen necessary for both the Oxo-process and the reduction was provided by the secondary alcohol, which in turn was converted into ketone.

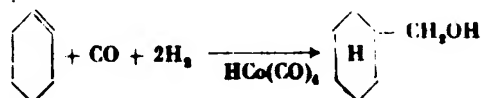
The importance of the direct alcohol synthesis consists in the fact that the reaction which has hitherto been carried out in two steps, can now be accomplished in a single operation. By means of this, the rather delicate reduction operation — especially with raw materials containing sulphur — can be avoided. From this point of view, however, the disadvantage of the 72% yield achieved by *I. Wender* and co-workers is that it makes separation very difficult. Yet, taking the advantages into consideration, it seemed useful to take up the direct alcohol synthesis and, first of all, to clarify the possibility of increasing the alcohol yield, in order to make the separation practicable, and by means of this to make the process possible on an industrial scale. In the course of our work carried out in the Hungarian Petroleum and Natural Gas Research Institute in connection with the Oxo-process, we, therefore, soon took up the investigation of the direct alcohol synthesis, this being in our opinion the most practicable way towards the realisation of the process.

In order to investigate the influence of temperature, the most important variable at the Oxo-process, three series of experiments have been carried out.

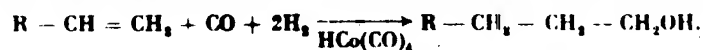
1. Homogeneous catalytic reduction of butyraldehyde:



2. Oxo synthesis and direct alcohol synthesis with cyclohexene :



3. Oxo synthesis and direct alcohol synthesis with cracked gasoline :



The experimental conditions as well as the results are shown in Tables 1, 2 and 3. For the reduction percentage with butyraldehyde see Figure 1. The reduction percentage and the quantity of gas reacted (mole gas for mole olefin) at the experiments with cyclohexene and cracked gasoline can be seen on Figures 2 and 3, respectively. The extent of the reduction was measured by the relative alcohol content of the oxygenated compounds, the aldehydes and alcohols, this value giving more characteristic results than the alcohol content of the end product in percents. The reason for this is that the removal of cobalt, which is necessary before the analysis, irreproducibly alters the absolute alcohol content, whereas the alcohol-aldehyde ratio does not change essentially. Therefore, the latter value is always shown in the figures. In the experiments with butyraldehyde the quantity of gas which reacts is not shown in the figure because, as can be seen on Table 1, this is invariably higher than is to be expected in accordance with the reduction. Obviously, some kind of a side reaction takes place, and this is still being investigated.

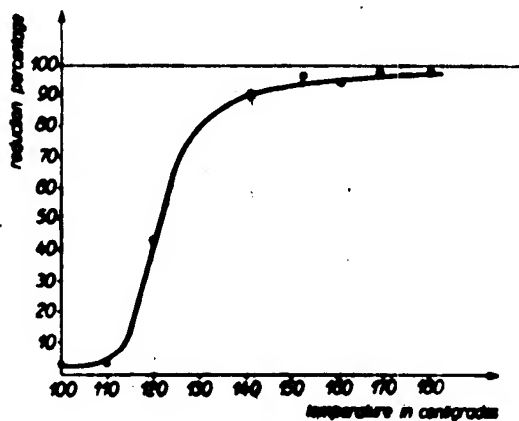


Fig. 1

Homogeneous catalytic reduction of butyraldehyde

Reduction percentage as a function of temperature

Catalyst : 3.2 mole per cent cobalt stearate, calculated for the aldehyde

Pressure : 160—200 atm.

Gas composition : 2 H₂+1 CO

Reaction time : 100 minutes

The following conclusions may be drawn from the diagrams:

Under the experimental conditions of the Oxo-process, n-butyraldehyde can be reduced at 150—155° C practically up to 100 per cent. This 100 per cent reduction, however, does not mean that the reaction product consists of butyl-

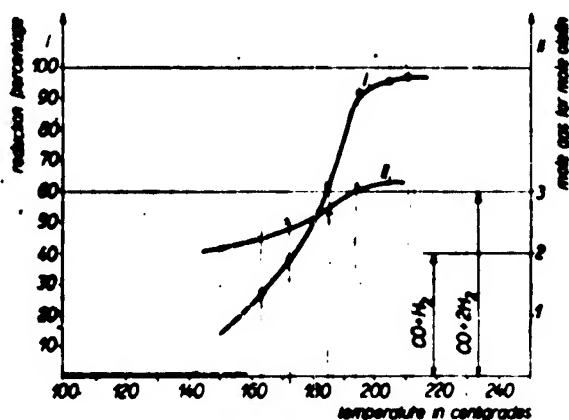


Fig. 2

Direct alcohol synthesis with cyclohexene

Reduction percentage (I) and gas consumption (II) as a function of the temperature

Catalyst: 0.8 mole percent cobalt stearate, calculated for the olefin

Pressure: 150—200 atm.

Gas composition: $2H_2 + 1 CO$

Reaction time: 30 minutes

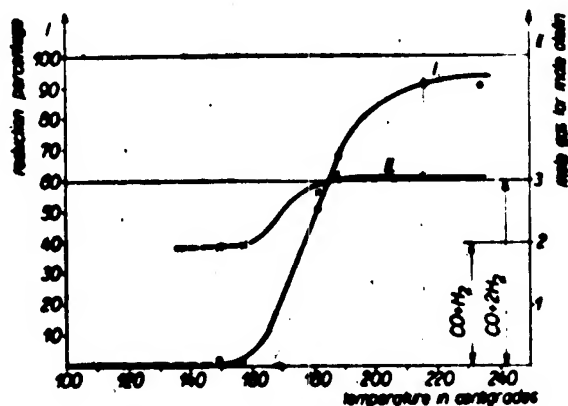


Fig. 3

Direct alcohol synthesis with cracked gasoline

Reduction percentage (I) and gas consumption (II) as a function of the temperature

Catalyst: 1.1 mole percent cobalt stearate, calculated for the olefin

Pressure: 130—200 atm.

Gas composition: $2H_2 + 1 CO$

Reaction time: 60 minutes

DIRECT SYNTHESIS OF ALCOHOLS FROM OLEFINS WITH A MIXTURE OF CARBON MONOXIDE AND HYDROGEN

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TABLE 1

Reduction experiments with butyraldehyde

Charge stock: 0.5 mole=36 g butyraldehyde in 110 ml motor spirit
 Catalyst: 3.2 mole % Co stearate calculated for the aldehyde
 Gas composition: $H_2:CO=2:1$
 Reaction time: 100 minutes
 Pressure: 160—200 atm.

No. of experiment	Temp. °C	Consumed gas in moles	End product		
			Aldehyde %	Alcohol %	Alcohol alcohol + aldehyde %
V. 14	100	0.61	7.04	0.33	4.1
V. 10	110—112	0.77	7.01	0.31	3.9
V. 13	120	0.61	8.72	6.60	43.0
V. 11	140—142	0.64	1.50	13.3	90.0
V. 17	149—155	1.47	0.64	17.5	96.4
V. 12	160	0.99	0.90	15.8	94.8
V. 16	167—170	1.00	0.29	17.8	98.0
V. 15	178—180	1.05	0.28	19.5	98.8

TABLE 2

Direct alcohol synthesis with cyclohexene

Charge stock: 0.2 moles=16.4 g cyclohexene in 400 ml motor spirit
 Catalyst: 0.8 mole % Co stearate calculated for olefin
 Gas composition: $H_2:CO=2:1$
 Reaction time: 30 minutes
 Pressure: 150—200 atm.

No. of experiment	Temp. °C	Consumed gas in moles	End product		
			Aldehyde	Alcohol	Alcohol alcohol + aldehyde %
V. 10a	100—109	—	—	—	—
V. 11a	138—147	—	—	—	—
V. 23	159—167	0.46	3.41	1.37	20.7
V. 11b	165—180	0.52	2.24	2.56	38.8
V. 76	185	0.54	1.32	2.73	62.3
V. 13	190—200	0.62	0.54	5.72	91.5
V. 14	203—206	0.83	0.38	8.10	95.6
V. 16	205—216	0.64	0.24	5.90	96.3

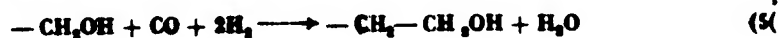
TABLE 3

*Direct alcohol synthesis with cracked gasoline**Charge stock: 37 g cracked gasoline containing 0.15 mole olefin in 370 ml motor spirit**Catalyst: 1.1 mole % Co stearate calculated for olefin**Gas composition: H₂: CO=2:1**Reaction time: 60 minutes**Pressure: 130—200 atm.*

No. of experiment	Temp. °C	Consumed gas in moles	End product		
			Aldehyde %	Alcohol %	Alcohol / alcohol + aldehyde %
V. 20	124—134	—	—	—	—
V. 21	146—155	0.30	4.75	0.11	2.3
V. 22	155—160	0.30	5.22	0.06	1.1
V. 17	179—185	0.42	2.87	3.06	51.5
V. 19	186—195	0.46	1.72	3.68	68.2
V. 24	214—222	0.47	0.47	4.97	91.5
V. 31	230—242	0.45	0.47	4.41	90.5

alcohol only, as C₈ and higher alcohols are also present. These higher alcohols are formed from higher aldehydes which in turn are formed from butyraldehyde as a result of aldol conversion. There are, however, hardly any carbonyl compounds in the end product.

The minimum temperature at which the formation of cobalt carbonyl and the Oxo-process started, respectively, was in the course of our direct synthesis experiments approximately 145° C. At this temperature practically no alcohol is produced, either with cyclohexene, or with cracked gasoline. By raising the temperature, the relative alcohol content increases, first slowly, then at a rapid rate, and reaches more than 95 per cent at 200—210° C with cyclohexene, and 90 per cent at 220° C with cracked gasoline. Alongside with the increase of the extent of reduction the gas quantity used in the individual experiments also increases from quantities in accordance with the Oxo-reaction, CO + H₂ (2 moles), to the quantity corresponding to the 100 per cent reduction, CO + 2H₂ (3 moles), and even somewhat above this. The gas consumption points to a more extensive reduction than borne out by the results. This latter fact may be caused by some side reaction in which additional gas is absorbed, possibly the homologue formation of the alcohols:



Comparison of the curves reveals that the reduction of higher aldehydes is carried out with greater difficulty than that of the lower members of the series.

It can be shown by means of approximate thermodynamic calculations [$\Delta G_{1500}^{\circ} + 0,8 \text{ Kcal/mole}$, $K_p = K_n \cdot P^{(\Delta n)} = 0,37$] that under such reaction circumstances equilibrium is shifted towards the formation of the alcohol practically to 100 per cent. It follows from the above that in the course of our experiments carried out for the same length of time no equilibrium was reached and the increased reduction at higher temperatures is due to increased reaction rates brought about by them. It is therefore probable that by an adequate increase in the duration of the reaction, an adequate reduction can be accomplished at lower temperatures as well. In spite of the above, the high temperature reaction is more interesting from the point of view of commercial realisation, due to better output. Corrosion of the materials of equipment must, however, be taken into consideration when applying higher temperature.

Further, we have investigated the correlation between the temperature and the total quantity of the oxygenated compounds formed. Certain investigators (*Roelen* [6]) are of the opinion that the greatest drawback to the direct alcohol synthesis consists in the olefins hydrogenating to paraffins due to the high temperatures required, without previously participating in the Oxo-process. Contrary to this opinion, in our experiments at higher temperatures, where conversion to alcohol was nearly complete, 3 moles gas were used for 1 mole olefin. Had a simple hydrogenation of the olefin taken place, the quantity of the consumed gas should have been much less, as only 1 mole gas is used for 1 mole olefin in case of hydrogenation. Consequently, no olefin saturation of importance took place in our experiments.

This assumption is also supported by the fact that in the liquid product the yield of oxygenated compounds calculated for the olefin was constant within the range of experimental and analytical errors.

The olefin saturation mentioned by certain investigators probably occurs under heterogeneous catalytic conditions. The absence of the saturation reaction of the olefins in our homogeneous catalytic reaction was to be expected, as, under these circumstances, the aldehyde formation in the Oxo-process is instantaneous and there is no time left for the hydrogenation of the olefin.

EXPERIMENTAL

The experiments were carried out in an electrically heated rocking autoclave of 870 ml. The necessary pressure was produced by means of a Hofer-type compressor. A thermocouple reaching into the autoclave was used for temperature measurement and the pressure recorded by a disk recording gauge.

The butyraldehyde was prepared from butyl alcohol by oxidation with potassium bichromate [7] and the cyclohexene from cyclohexanol by dehydration with sulphuric acid [8]. Both materials were fractionated in a Widmer column. The purity of the products exceeded 96 per cent in every experiment. The data of the cracked gasoline were as follows:

Initial boiling point	31° C	Composition :	28% aromatics
End boiling point	147° C		40% olefins
Average molecular weight	100		30% paraffins-naphthenes
No. of carbon atoms	C_6-C_{10}	Spec. gravity	0.7214

The catalyst consisted exclusively of cobalt stearate and no carrier or metallic cobalt was used. The cobalt stearate was produced from cobalt acetate by fusing it with the equivalent quantity of stearic acid and subsequently distilling off the acetic acid and crystal water. Cobalt stearate is easily soluble in organic solvents, like gasoline, and its application is therefore rather simple.

Direct alcohol synthesis with cyclohexene

14.6 g cyclohexene (0.20 mole) and 1 g cobalt stearate were dissolved in 400 ml motor spirit and placed into the autoclave. The strong dilution of the olefin served the purpose to keep the temperature of the reaction mixture practically constant in spite of the high reaction rate and exothermic character of the Oxo-process. Before using such highly diluted mixtures it often occurred that due to the large amount of heat set free the temperature was raised even by 50° C, as a result of which the experiments became irreproducible. The dilution does not interfere with the smooth course of the reaction.

The autoclave was charged to 40 atm. with carbon monoxide and then to 120 atm. with hydrogen, which corresponds to about 2.2 moles gas, equivalent to a fourfold excess. The autoclave was then heated to the necessary temperature without shaking. Shaking was only started when the temperature had become constant. Generally a very quick, almost instantaneous pressure drop took place, and the consumed gas was somewhat in excess of the quantity necessary for the aldehyde formation. The shaking was continued for 30 more minutes, during which period the pressure drop was much slower. Shaking was then discontinued and the apparatus cooled. The pressure was again recorded at room temperature, and the amount of gas consumed by the reaction calculated from the difference between the starting and the end pressures.

The colour of the end product ranged from yellow to dark brownish red but it became much darker in all cases after storage in air. This is probably due to the gradual conversion of the colourless cobalt carbonyl hydride to cobalt carbonyl.

Before carrying out the analysis, the dissolved cobalt compounds had to be removed from the end product, because they interfere with the determination. This was achieved by stirring with 5% sulphuric acid at 80° C, when the cobalt compounds were converted into cobalt sulphate, which imparts a pink colour to the sulphuric acid solution. After separation and drying with lime hydrate the end product is ready for analysis. The determination of the carbonyl compounds was carried out by the hydroxylamine chlorohydrate method[9], and that of the alcohols with phthalic anhydride[10]. The advantage of the latter as against the acetic anhydride method is that it is not influenced by aldehydes.

We wish to express our thanks to Director M. Fround for his valuable assistance and to the laboratory personnel whose cooperation made it possible to compile the above data.

SUMMARY

On the basis of the above described experiments, increasing the temperature of the Oxo-process resulted in the synthesis of alcohols at 96 per cent yields using cyclohexene and 90 per cent yields with cracked gasoline by means of the direct alcohol synthesis. During the reduction carried out simultaneously with the Oxo-process the olefins were not hydrogenated to paraffins and the conversion to oxygenated compounds was close to 100 per cent.

Thus the formation of alcohols from olefins by direct alcohol synthesis has been made practically complete and the process is ready for development on an industrial scale.

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ПРЯМОЙ СИНТЕЗ АЛКОГОЛЕЙ ИЗ ОЛЕФИНОВ ПРИ ПОМОЩИ ГАЗОВОЙ СМЕСИ ОКИСИ УГЛЕРОДА И ВОДОРОДА

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Резюме

В процессе работы над оксосинтезом гомогенной фазы мы исследовали, какое влияние оказывает изменение температуры с одной стороны на однородное каталитическое восстановление бутиральдегида, а с другой на оксосинтез циклогексена и крекингбензина. Нами установлено, что бутиральдегид при температуре 150-155° С может быть практически восстановлен полностью, т. е. на 100%-ов. Затем установлено, что из циклогексена при температуре 200-210° С в один прием можно получить 95%-ный выход спирта, а из крекингбензина при температуре 200° С 90%-ный выход.

Далее нами установлено, что с повышением температуры не снижается процент использования исходного материала, т. е. олефины не гидрогенизируются в парафины. Следовательно, этот прямой синтез спиртов может быть осуществлен как с технологической, так и с экономической точек зрения. В случае переработки содержащих серу олефинов ввиду возможности избежать необходимости применения довольно тонкого процесса контактно-каталитического восстановления он может быть легче осуществлен, чем первоначальный двухступенчатый процесс.

UNMITTELBARE SYNTHES E VON ALKOHOLEN AUS OLEFINEN MITTELS EINES GASGEMISCHES AUS KOHLENMONOXYD UND WASSERSTOFF

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ZUSAMMENFASSUNG

Im Laufe ihrer Arbeit untersuchten die Verfasser, welchen Einfluss die Änderung der Temperatur einerseits auf die homogene katalytische Reduktion von Butyraldehyd mittels eines Gemisches von Kohlenmonoxyd und Wasserstoff andererseits auf die Oxydation mit Cyklohexen und Krackbenzin ausübt. Es wurde festgestellt, dass sich Butyraldehyd bei 150-155° C praktisch 100%-ig reduzieren lässt, sowie dass sich aus Cyklohexen bei 200-210° C mit einer Ausbeute von 95% und aus Krackbenzin bei 220° C mit einer Ausbeute von 90% Alkohole in einer einzigen Operation herstellen lassen.

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Die Verfasser haben nachgewiesen, dass bei Erhöhung der Temperatur die auf das Olefin berechnete Ausbeute nicht abnimmt, d. h., dass die Olefine nicht zu Paraffinen hydriert werden. Diese direkte Alkoholsynthese kann daher sowohl technisch, als auch wirtschaftlich gleich gut ausgeführt werden und lässt sich im Falle der Verarbeitung von schwefelhaltigen Olefinen leichter verwirklichen, als das ursprüngliche zweistufige Verfahren, da hier die heikle Operation der kontaktkatalytischen Reduktion vermieden wird.

József Berty, Magyar Ásványolaj és Földgáz-Kísérleti Intézet, Veszprém

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